

# The atomic scale investigation of Nb for superconducting RF cavity

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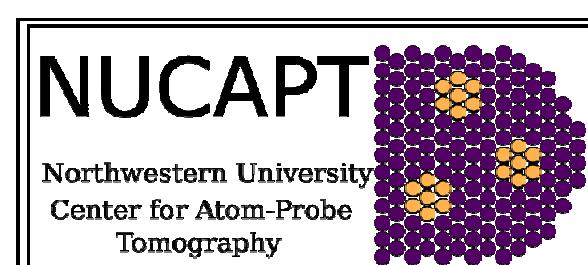
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- Fermi Lab

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- ANL



# Outline

- Oxygen in Nb
- Sample preparation
- Atom Probe tomography (APT)
- Result from Local Electrode Atom Probe (LEAP) tomograph
- Conclusions
- Next step

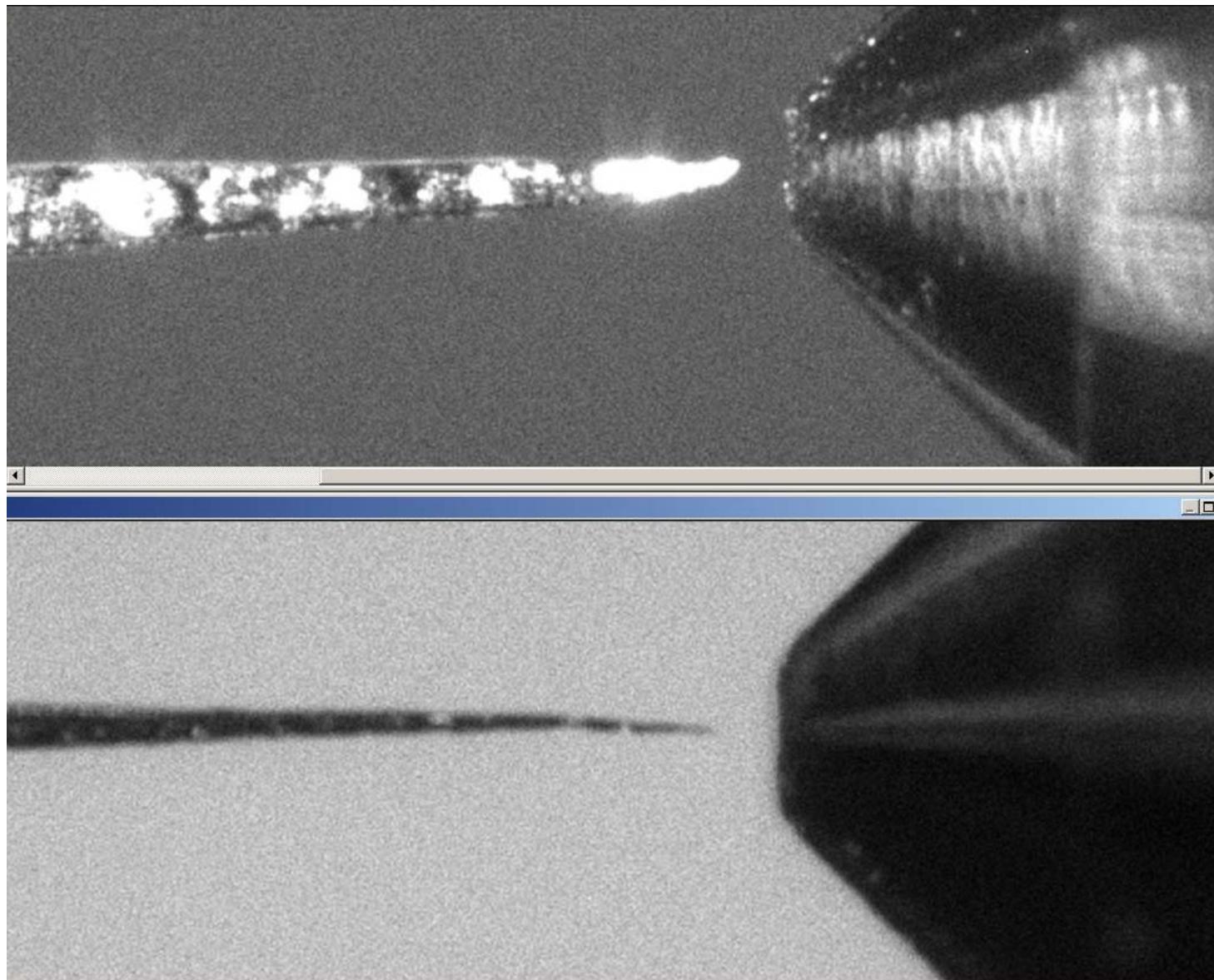
# Nb Superconducting RF cavity

- Performance of Nb cavity
  - Oxygen
  - Hydrogen
- Oxide on surface
  - Chemistry, thickness
- Oxygen in bulk
  - Concentration, location, and form

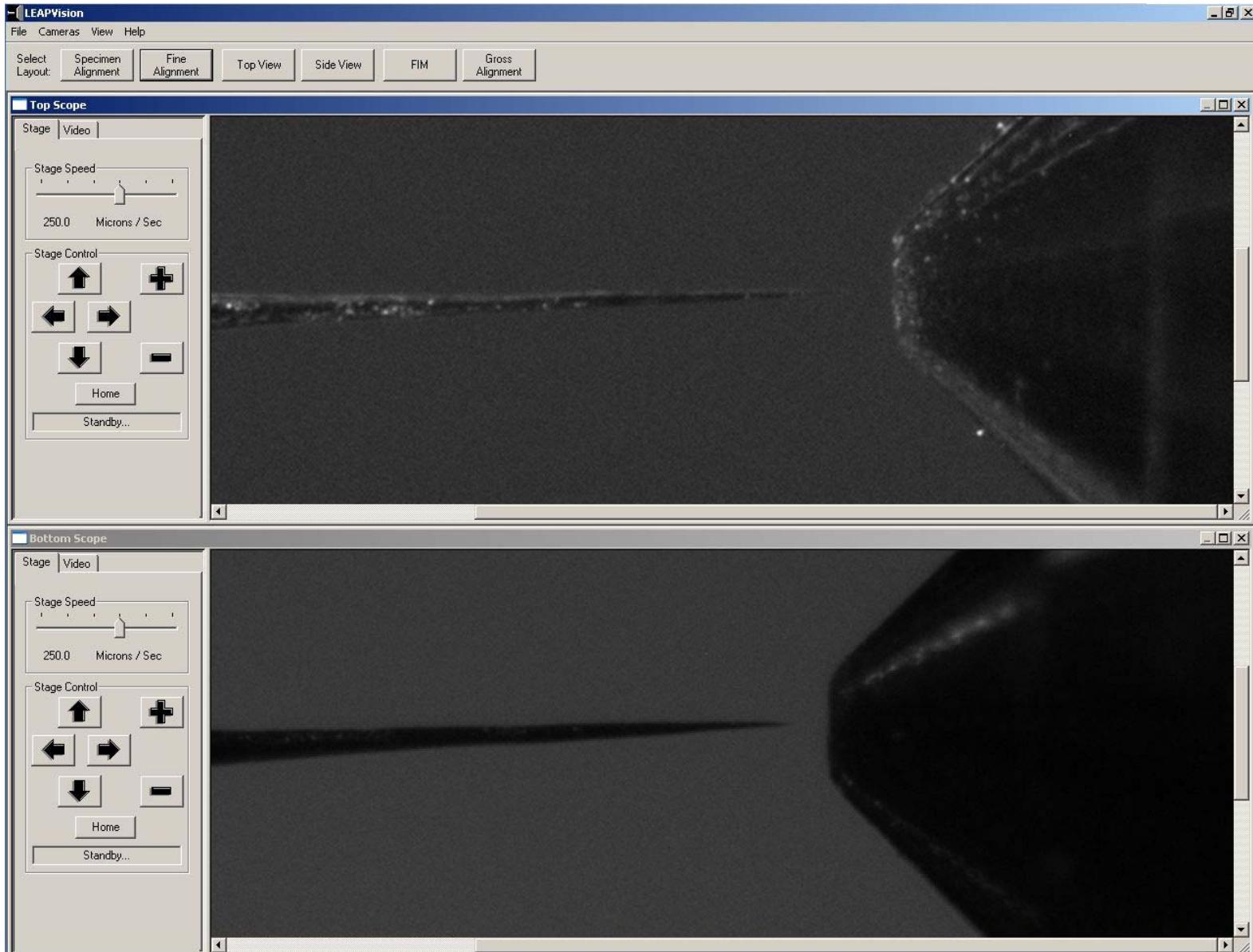
# Sample preparation

- $0.5 \times 0.6 \times 10 \text{ mm}^3$  Nb blanks
- $0.2 \times 0.3 \times 10 \text{ mm}^3$  Nb blanks
- $0.2 \times 0.2 \times 10 \text{ mm}^3$  Nb blanks
- Electropolishing
- HF :  $\text{H}_2\text{SO}_4 = 1 : 10$ 
  - With a few drops of  $\text{HNO}_3$
- Use DC voltage
  - Start at 31 V ~ decrease to 10 V
  - Minimize pulsing
- Rinse with deionized water

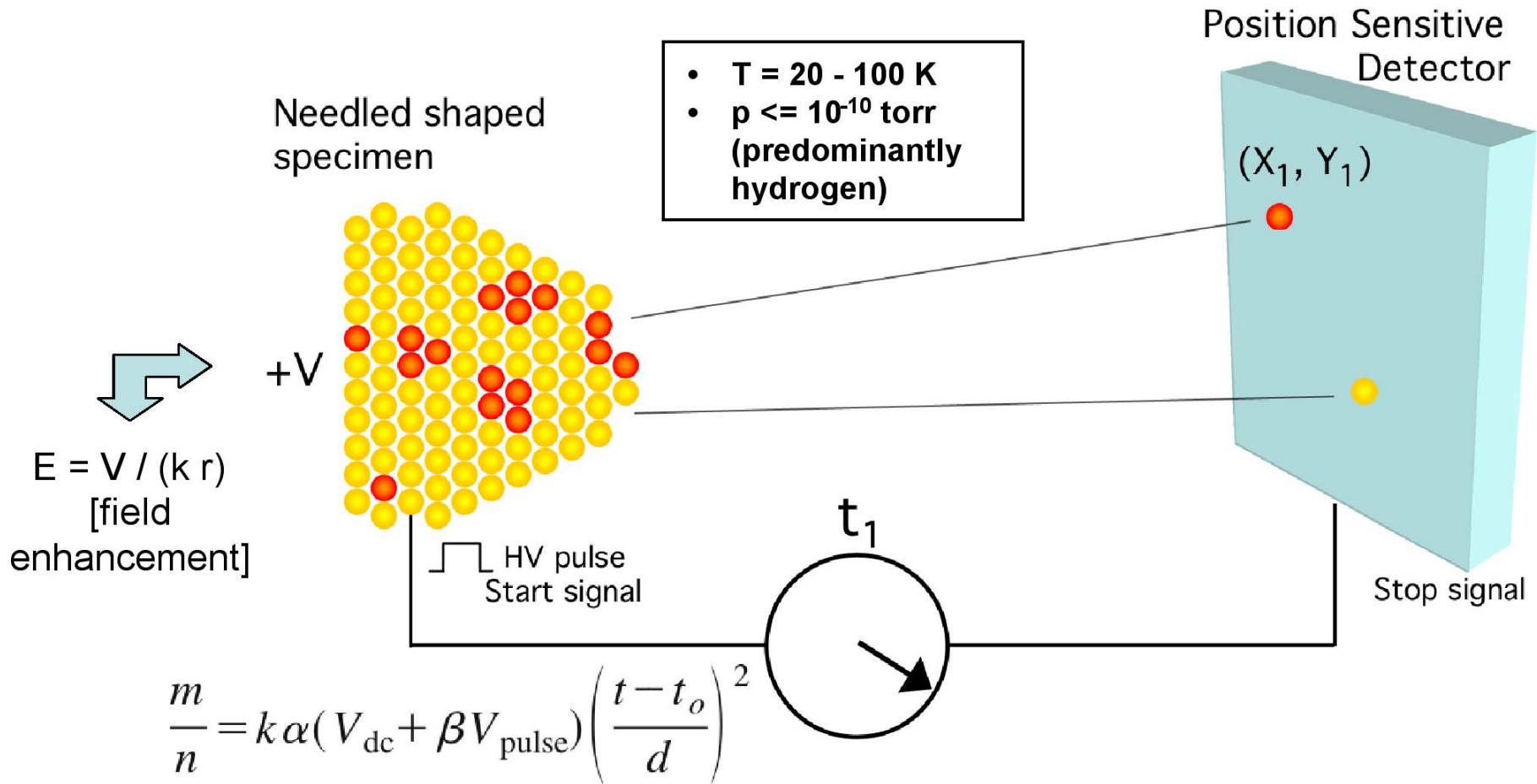
# Nb tip after EP 1



# Nb tip after EP 2



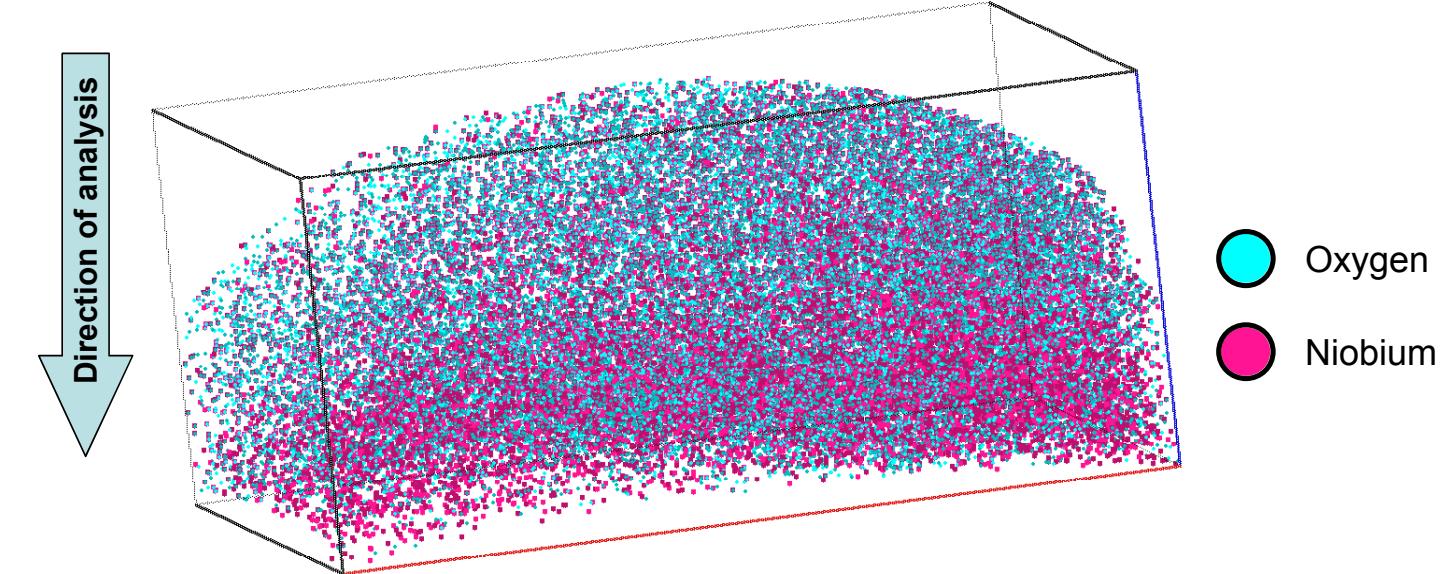
# Atom-probe tomography (APT)



- Coordination of ions (x, y, and z): Allows for the three-dimensional reconstruction of real space
- times-of-flight: Mass-to-charge ratio= identifying the elements

$$neV_0 = \frac{1}{2} m \frac{d^2}{t^2} \quad \frac{m}{n} = 2eV_0 \frac{t^2}{d^2}$$

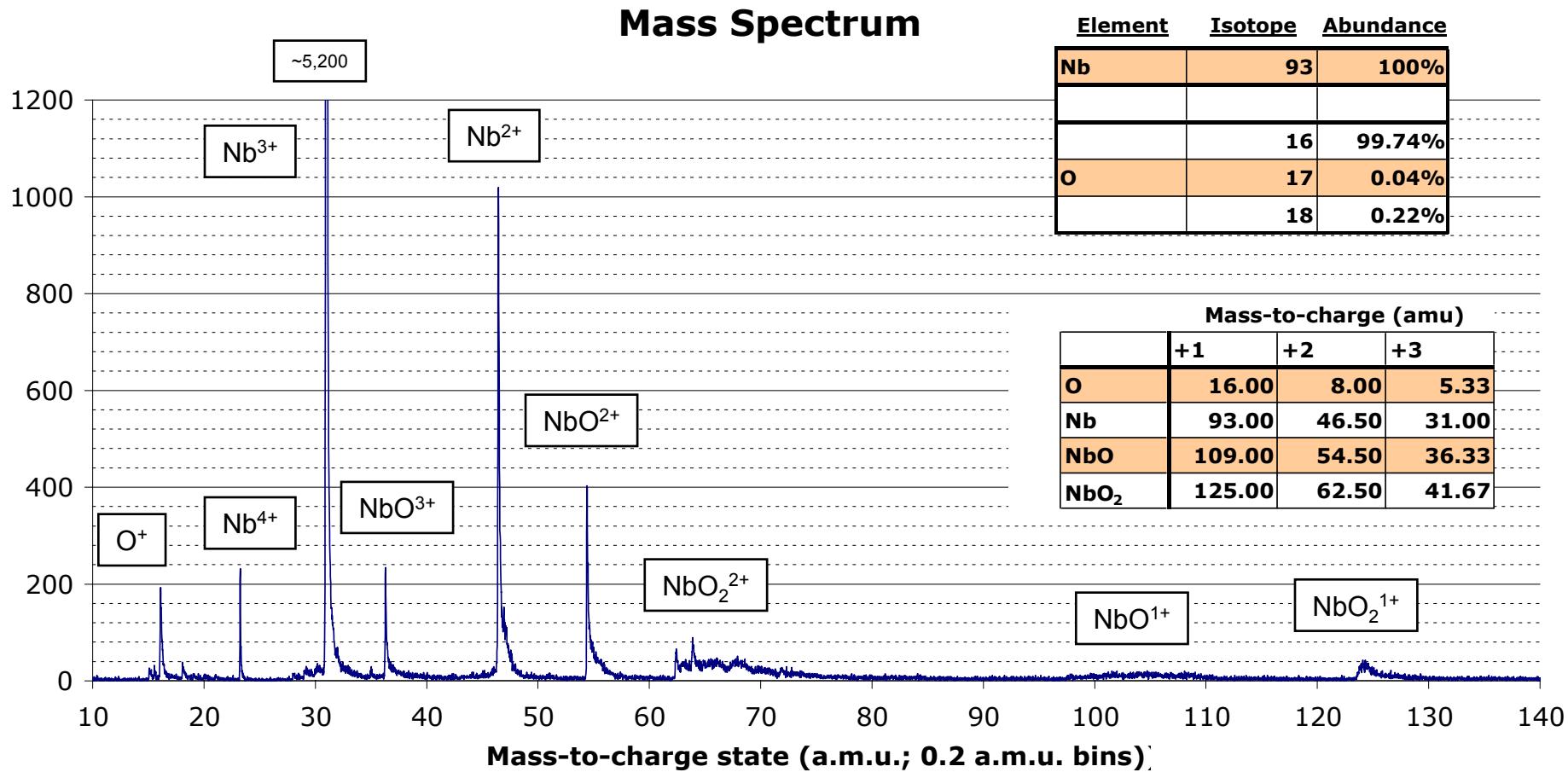
- Atomic resolution



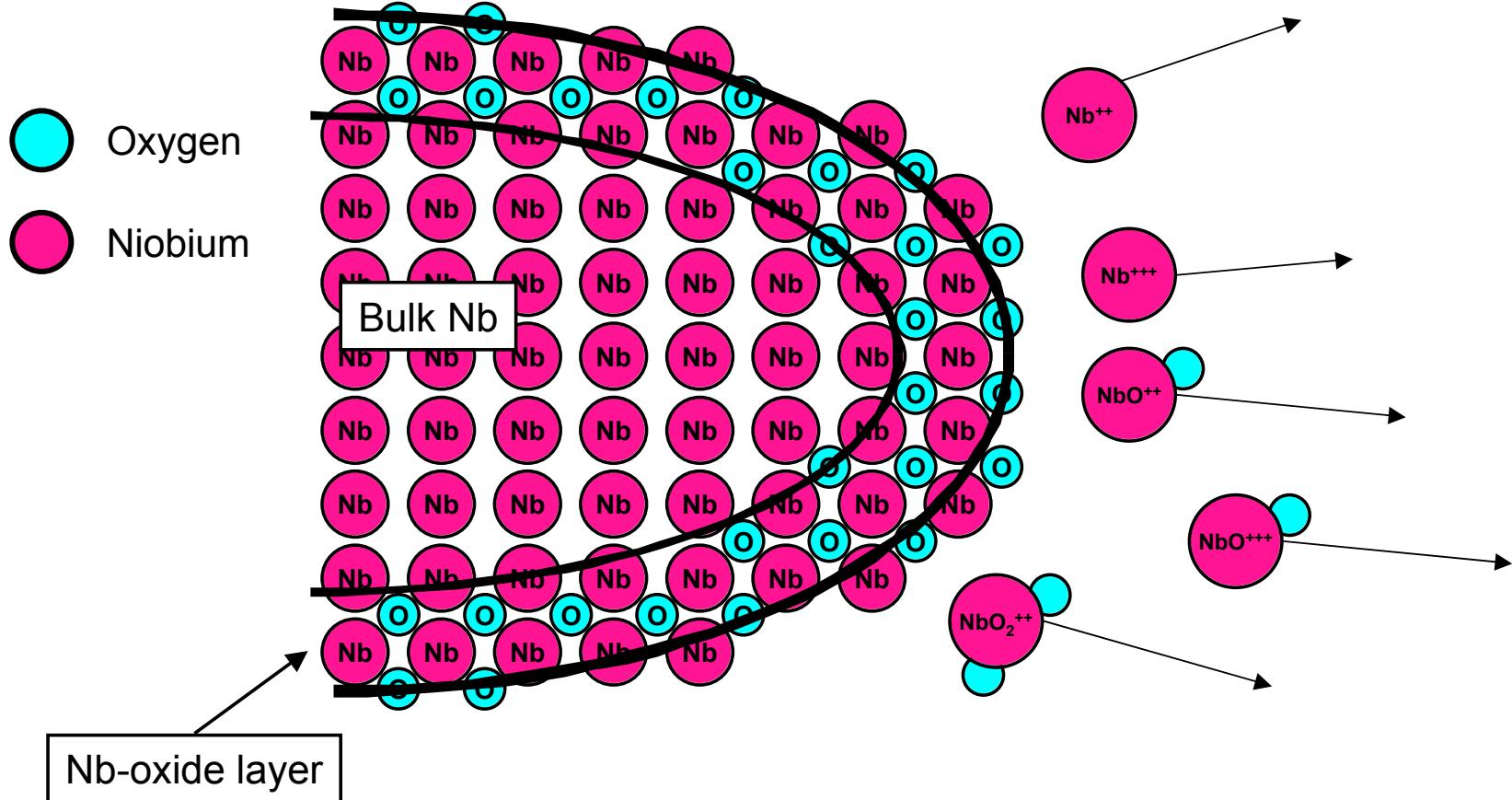
# Previous Results

- $23 \times 21 \times 11 \text{ nm}^3$
- 112 K atoms
- Nb magenta
- O cyan

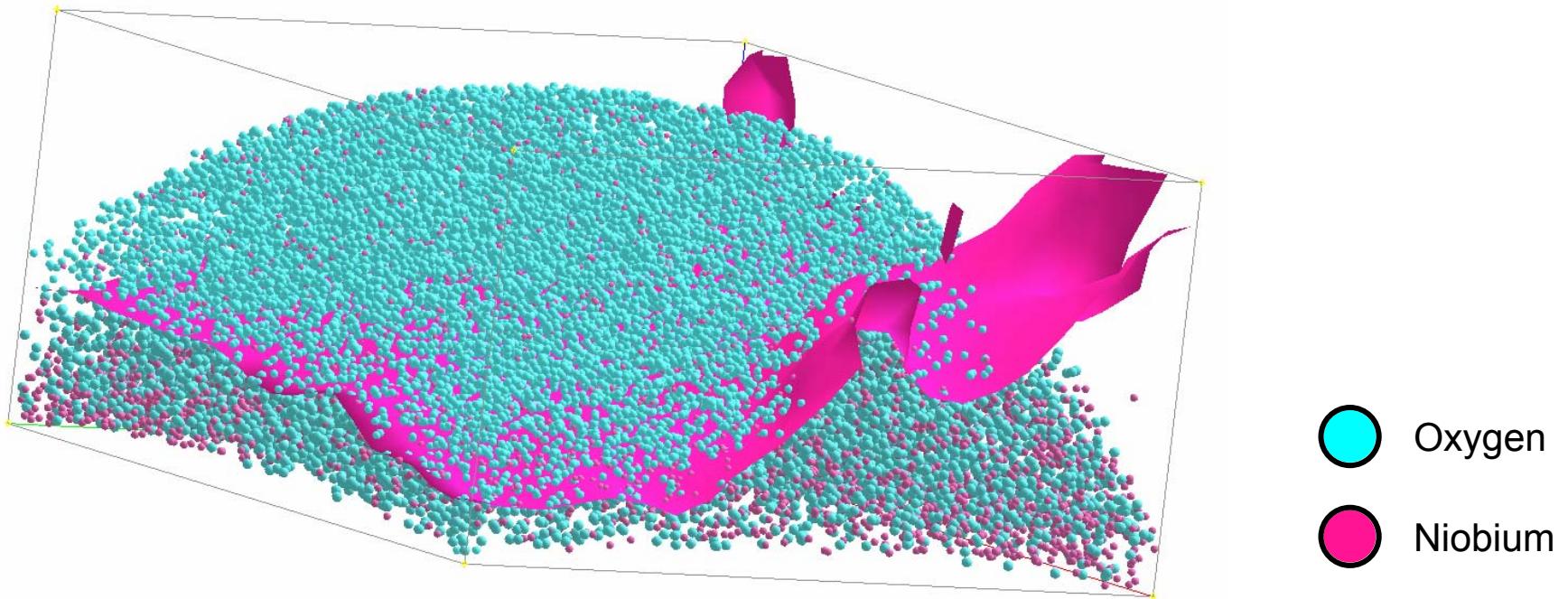
# Mass spectrum



# Evaporation of complex ions

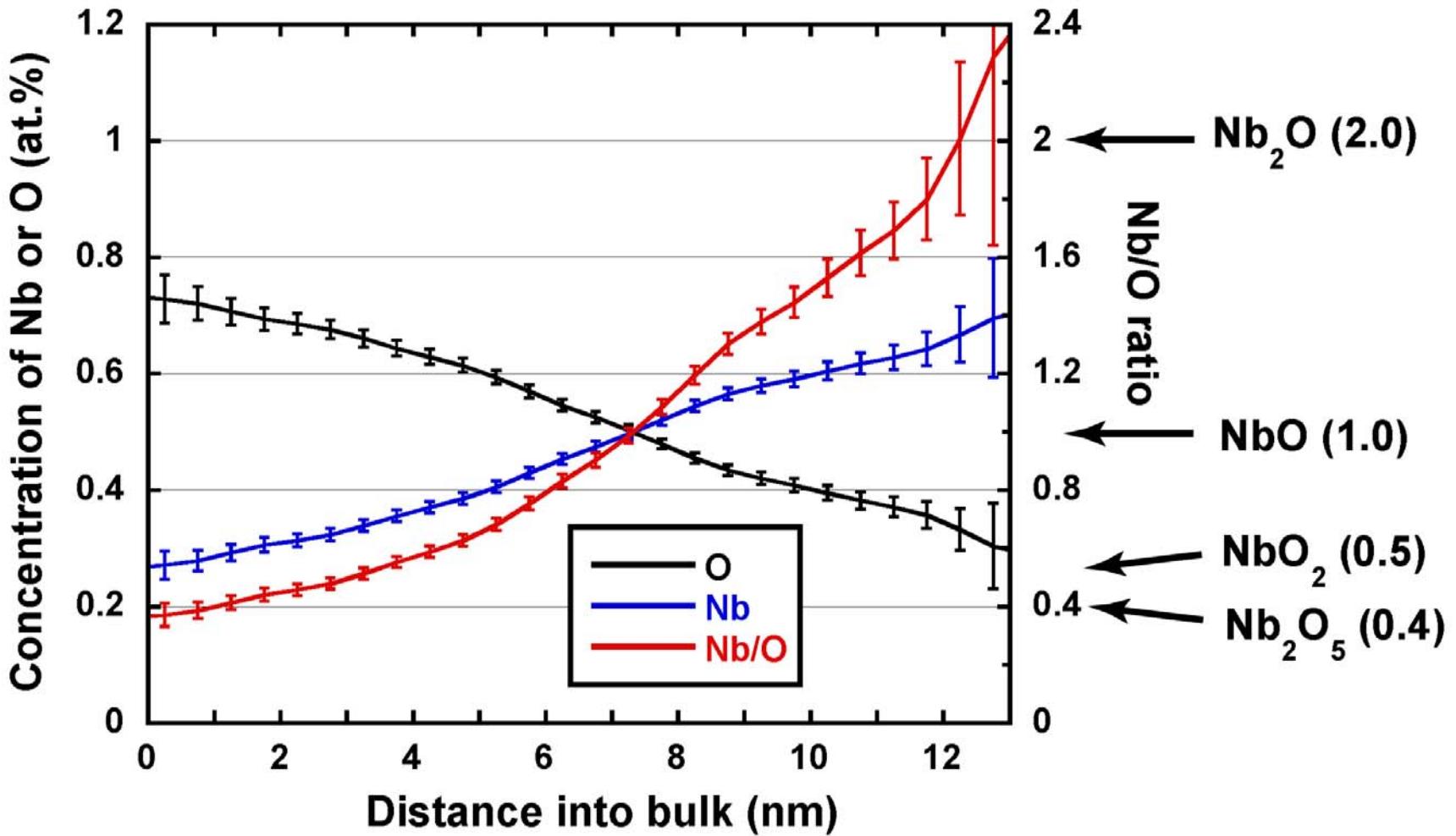


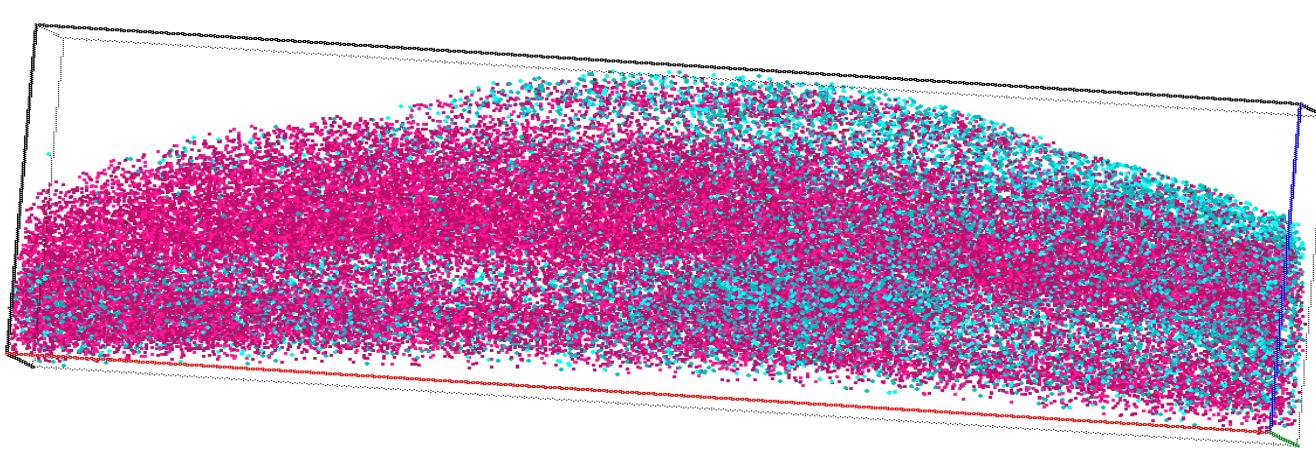
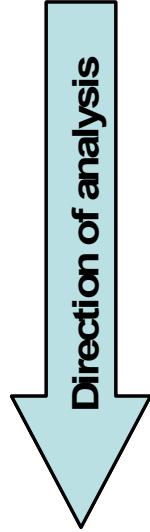
# Isoconcentration surface



- Identify the interface
- 30 at.% Nb isocentration surface

# Proximity Histogram

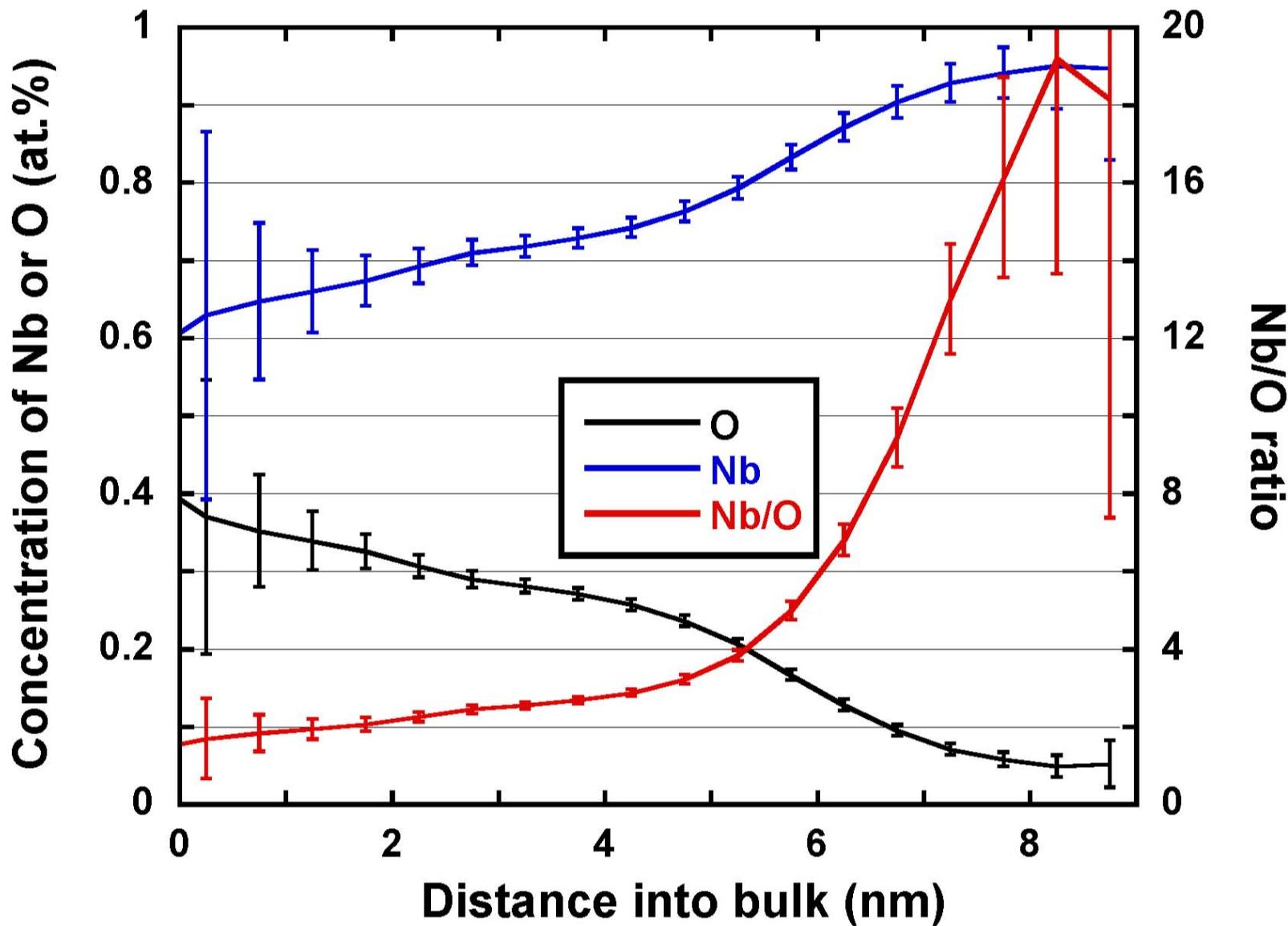




# Previous Results

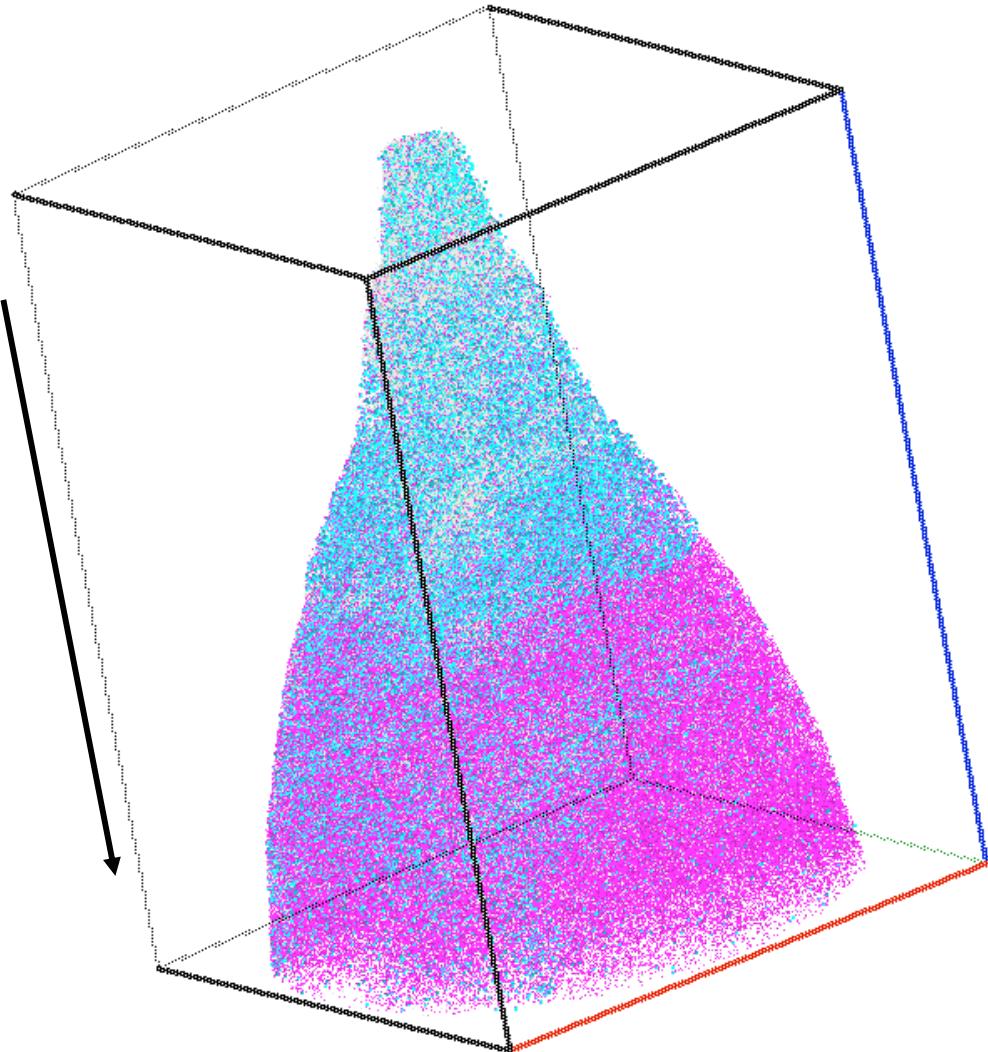
- $25 \times 25 \times 6 \text{ nm}^3$
- 75 K atoms
- Nb magenta
- O cyan

# Proximity Histogram



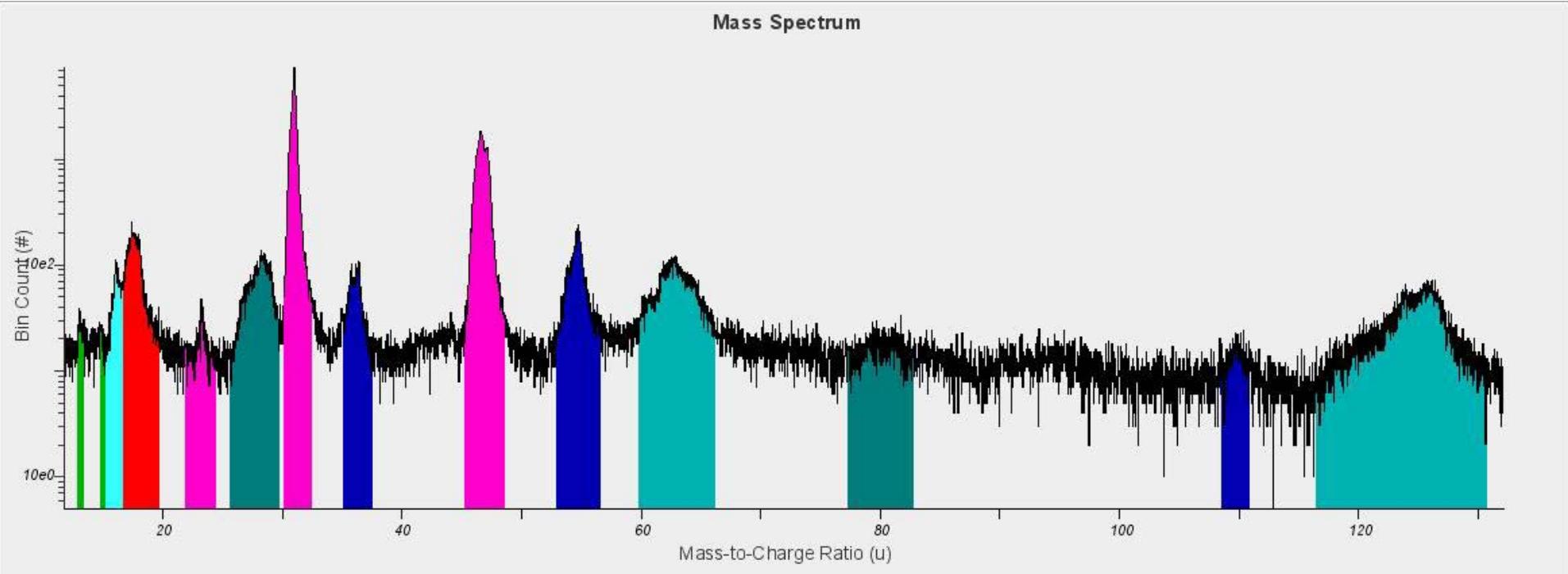
# 1<sup>st</sup> Results

- $25 \times 26 \times 48 \text{ nm}^3$
- 0.5 M atoms
- Nb magenta
- O cyan



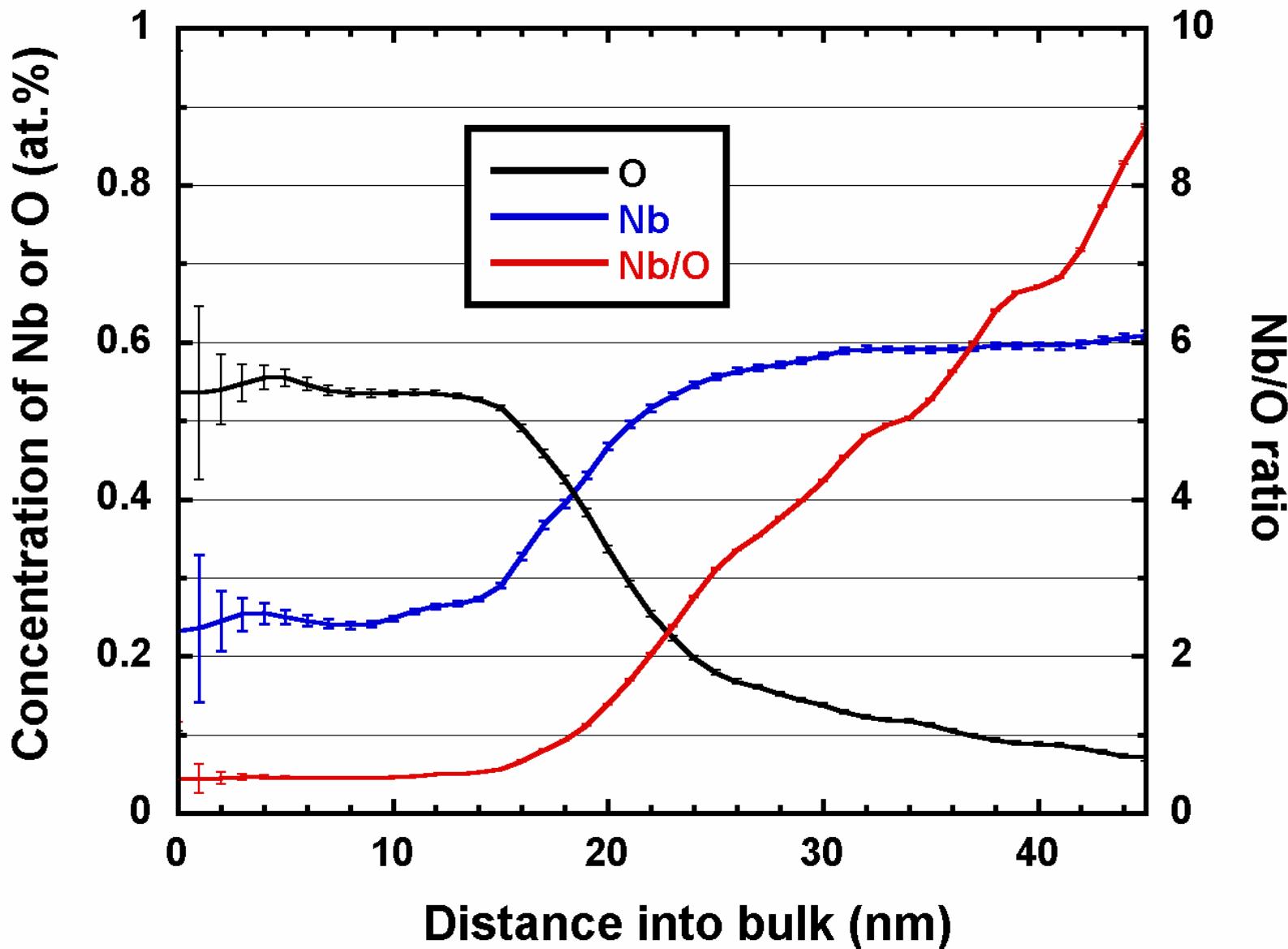
Analysis  
direction

# Mass spectrum



- $\text{NH}^+$ : 15  $\text{O}^+$ : 16  $\text{H}_2\text{O}^+$ : 18
- $\text{Nb}^{4+}$ : 23.25  $\text{NbOH}_6^{4+}/\text{NbO}_3\text{H}_3^{5+}$ : 29
- $\text{Nb}^{3+}$ : 31  $\text{NbO}^{3+}$ : 36.3
- $\text{Nb}^{2+}$ : 46.5  $\text{NbO}^{2+}$ : 54.5  $\text{NbO}_2^{2+}$ : 62.5
- $\text{NbO}^+$ : 109  $\text{NbO}_2^+$ : 125

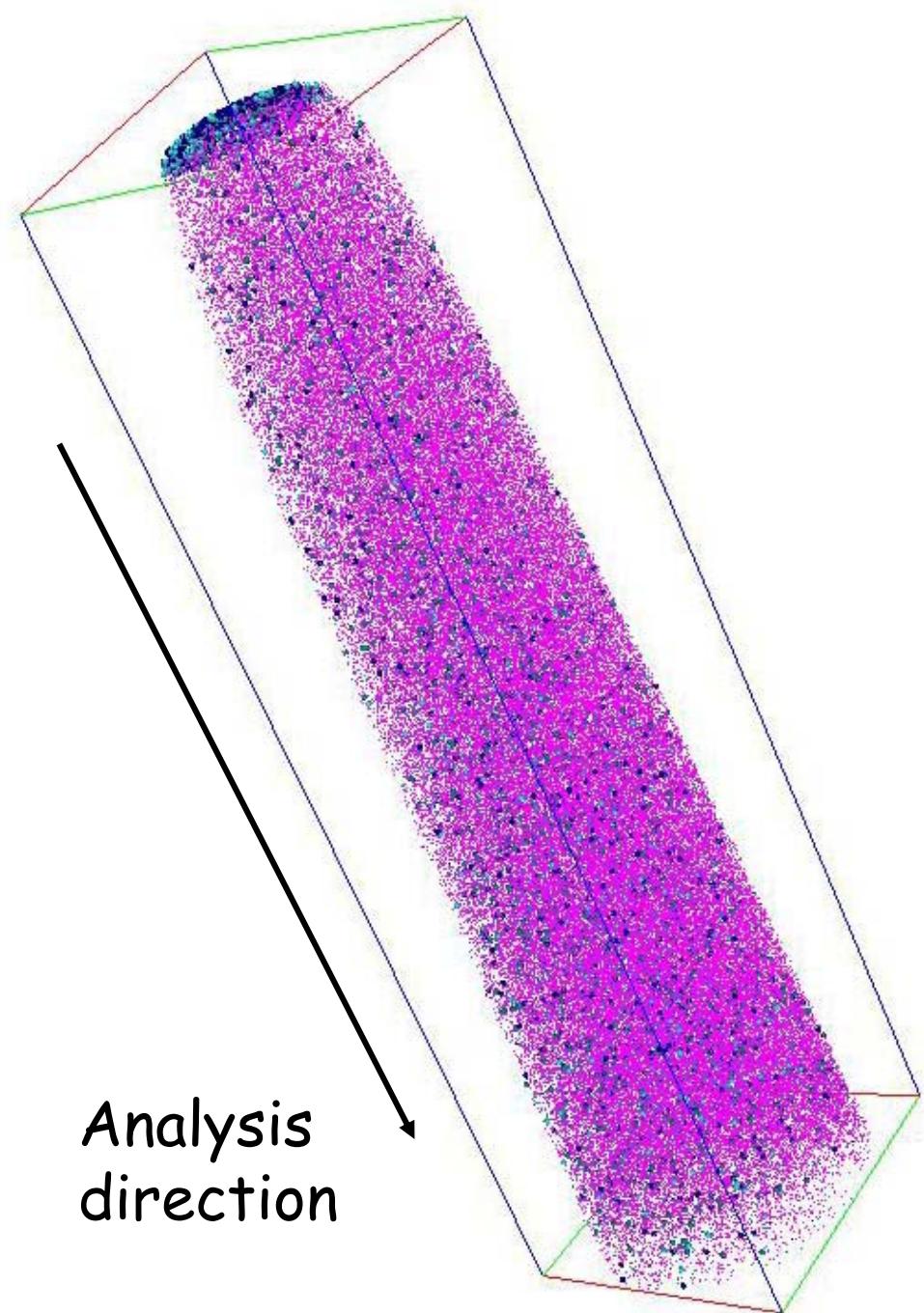
# Proximity Histogram



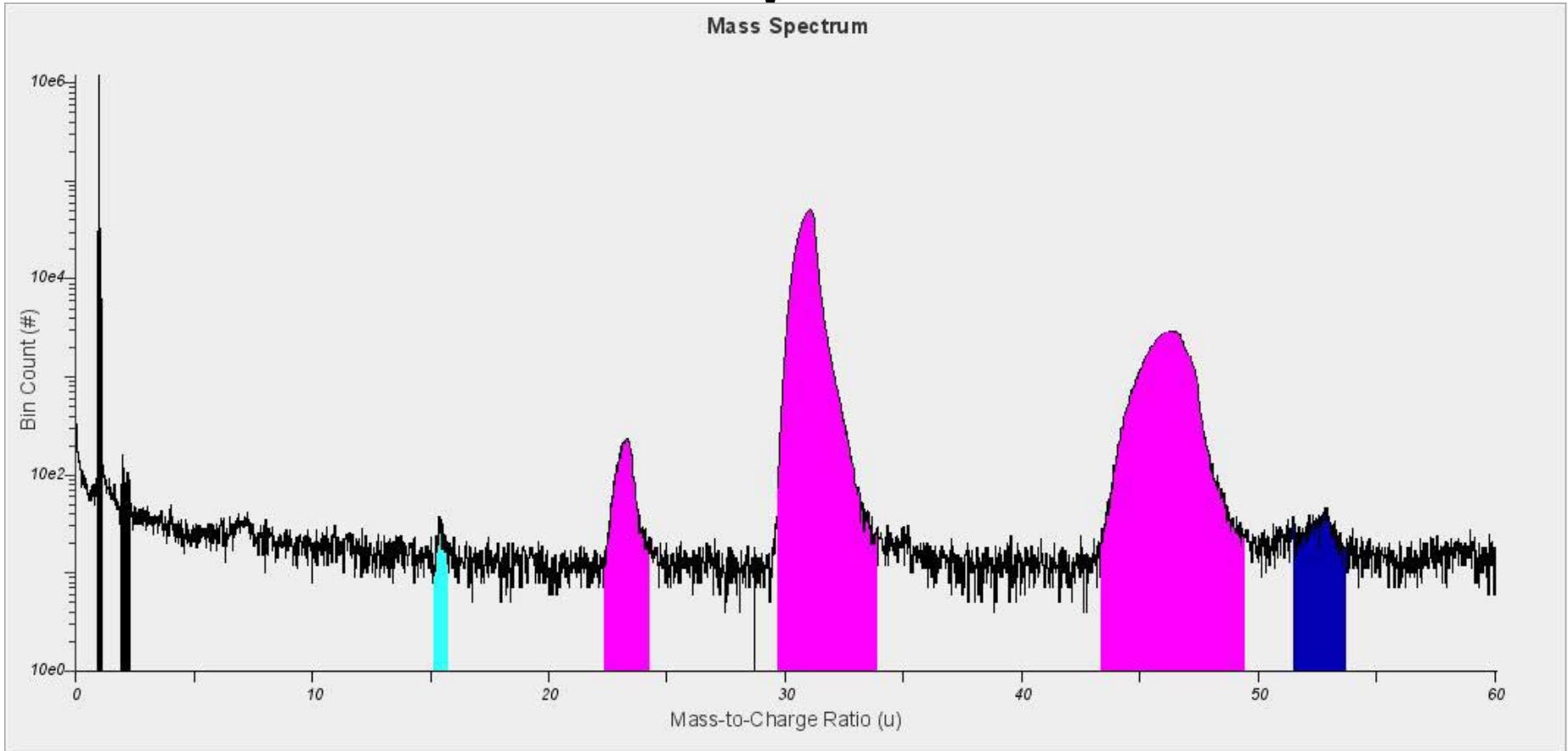
## 2<sup>nd</sup> Results

- $38 \times 40 \times 145 \text{ nm}^3$
- 3.8 M atoms
- Nb magenta
- O cyan

Analysis  
direction

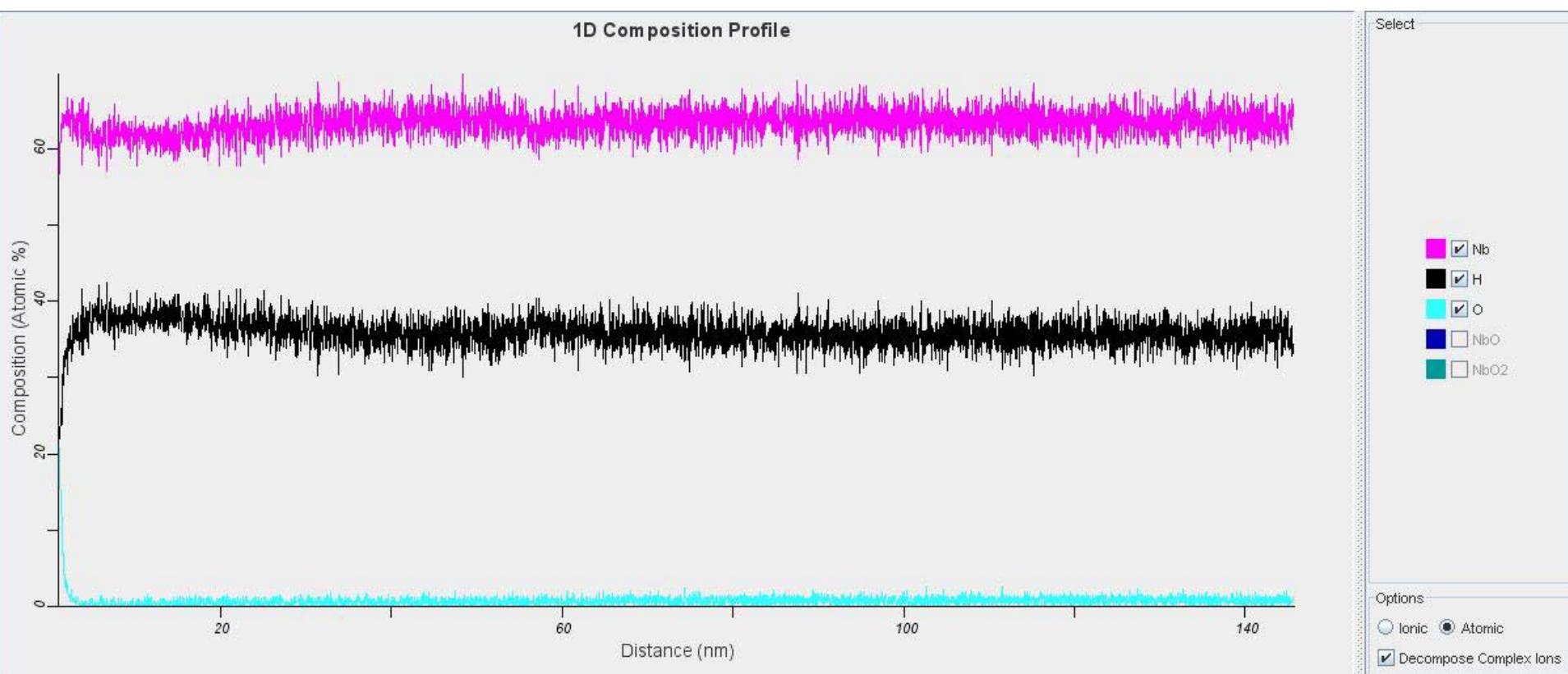


# Mass spectrum

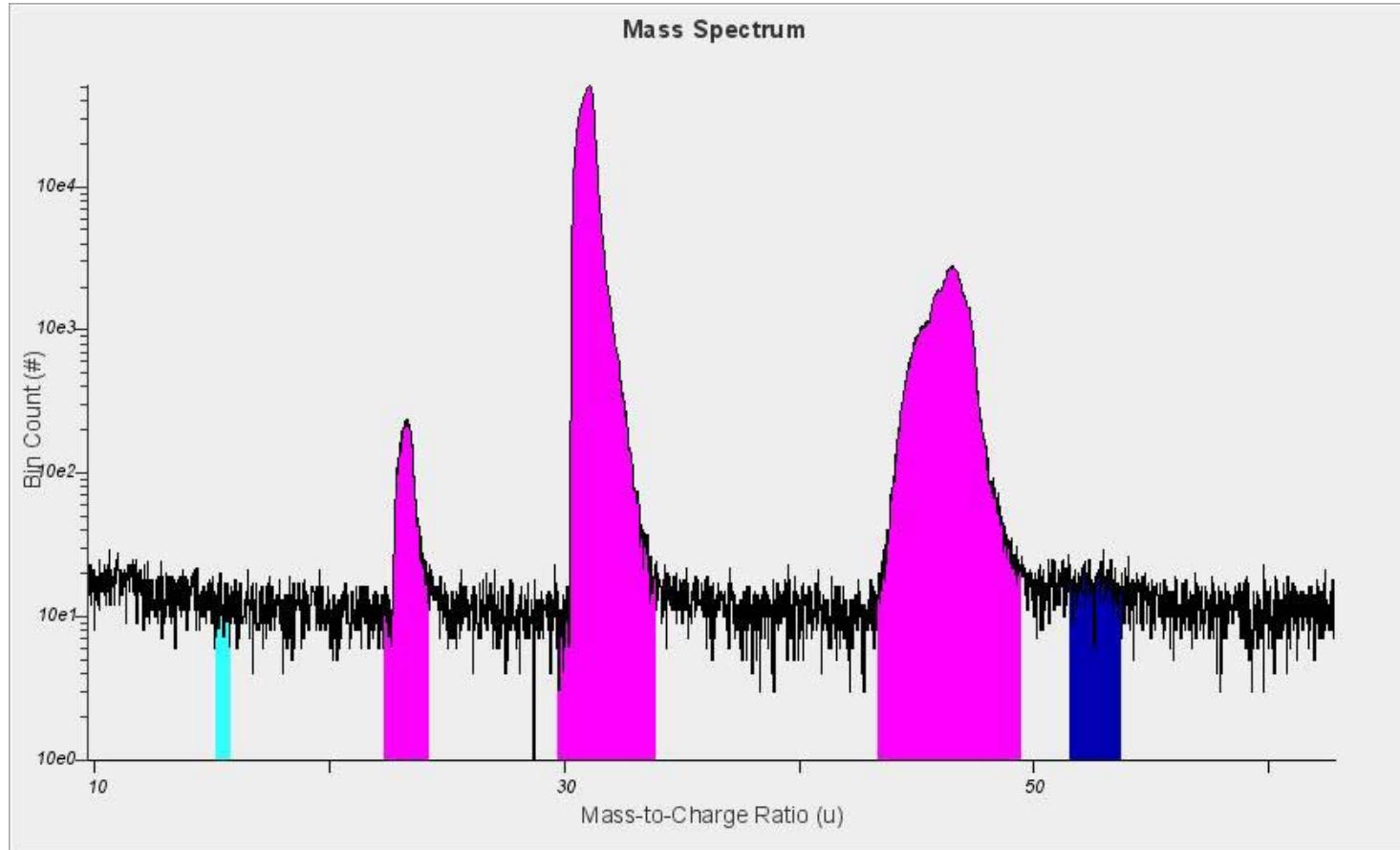


- $O^+$ : 16  $Nb^{4+}$ : 23.25  $Nb^{3+}$ : 31
- $Nb^{2+}$ : 46.5  $NbO^{2+}$ : 54.5

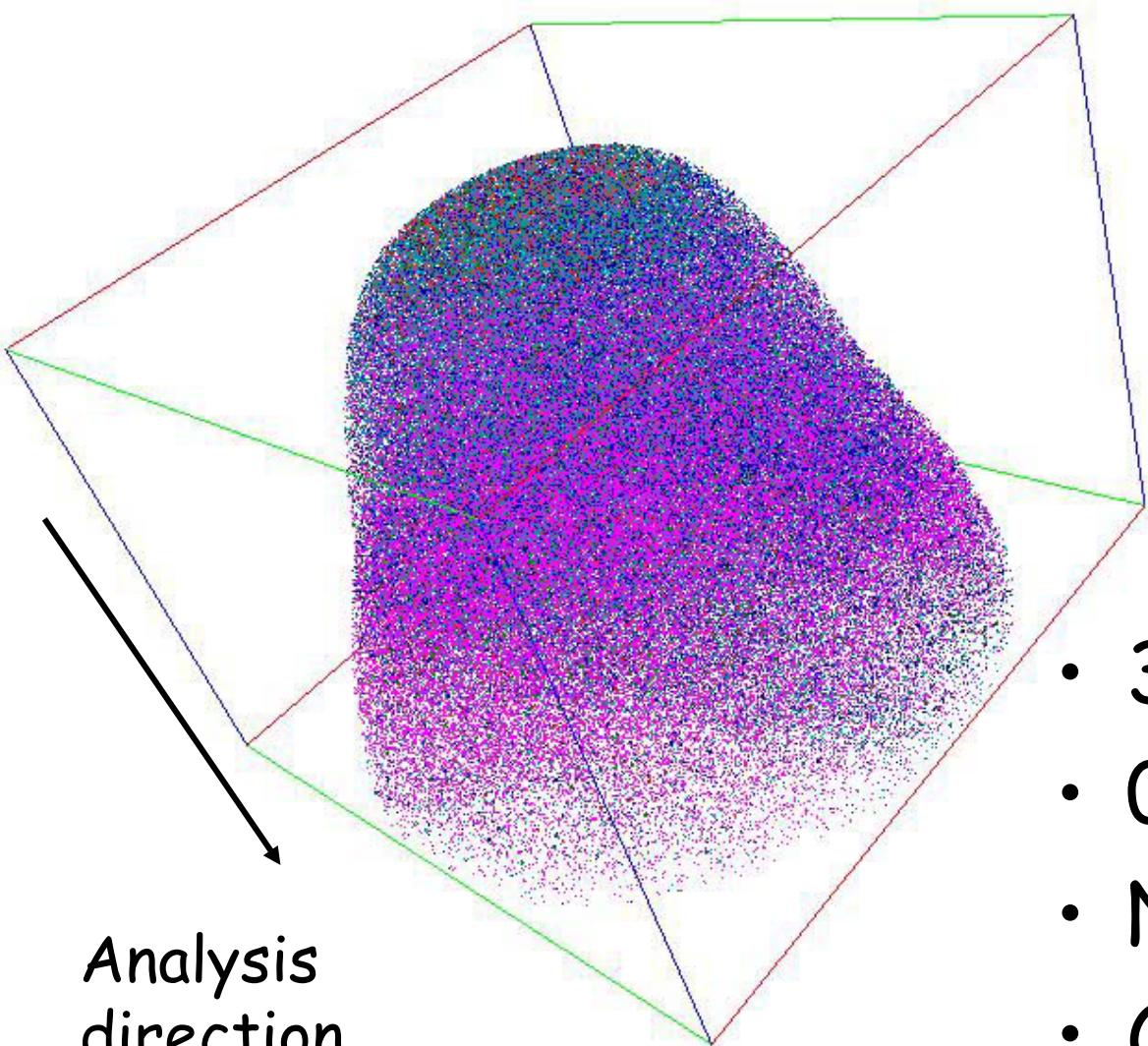
# Composition profile



# Mass spectrum of bulk



- No oxygen peak
- Less than 50 ppm



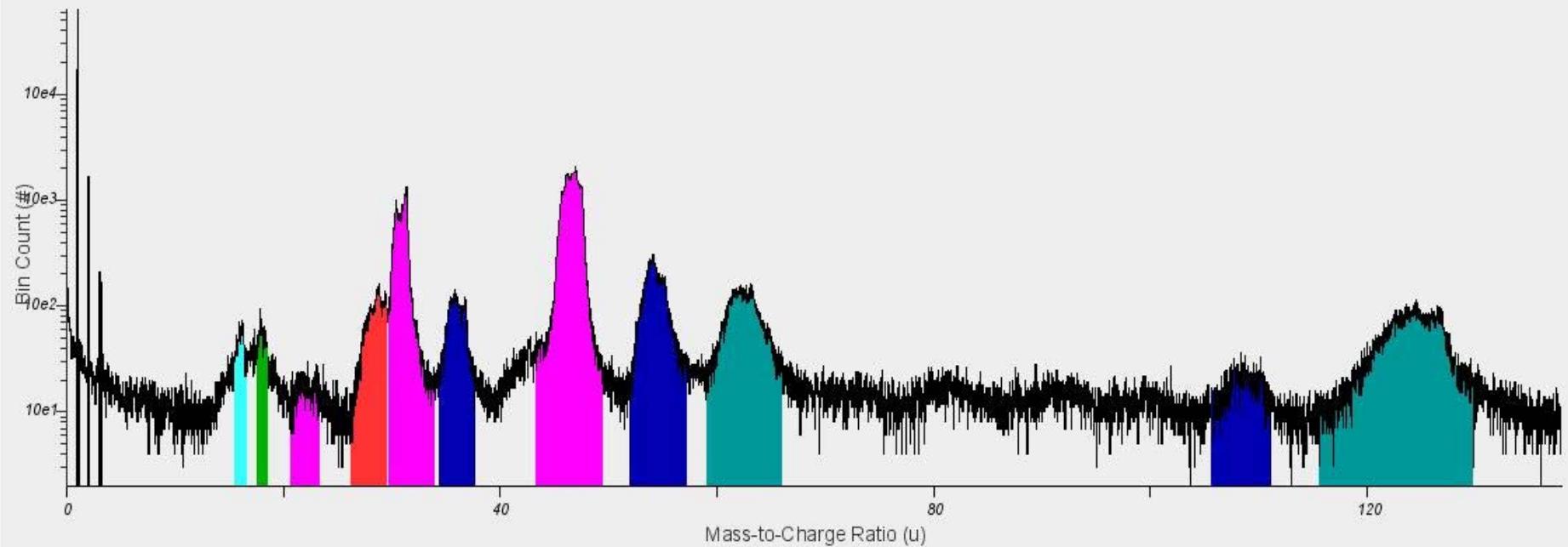
## 3<sup>rd</sup> Results

- $36 \times 35 \times 26 \text{ nm}^3$
- 0.5 M atoms
- Nb magenta
- O cyan

Analysis  
direction

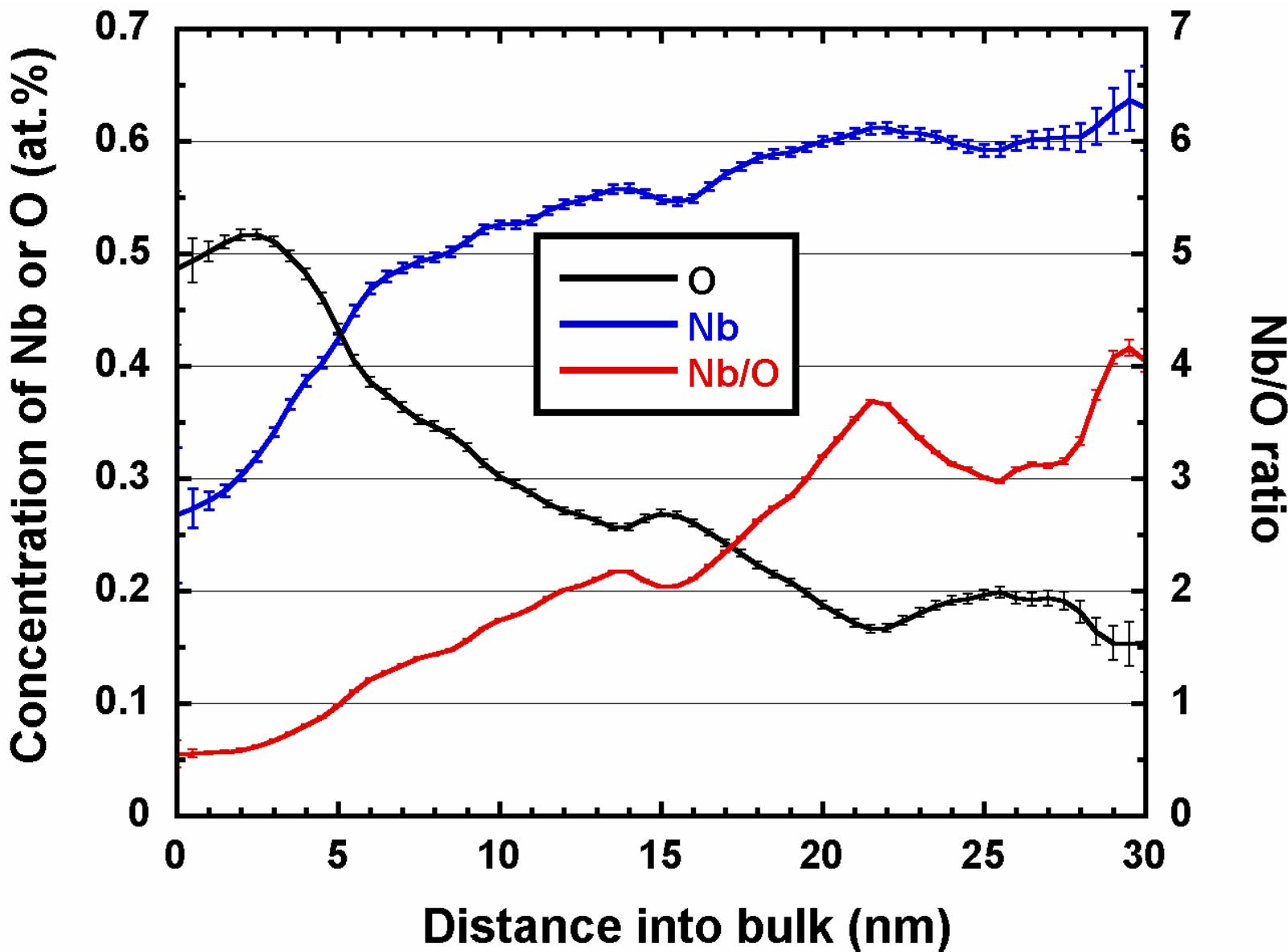
# Mass spectrum

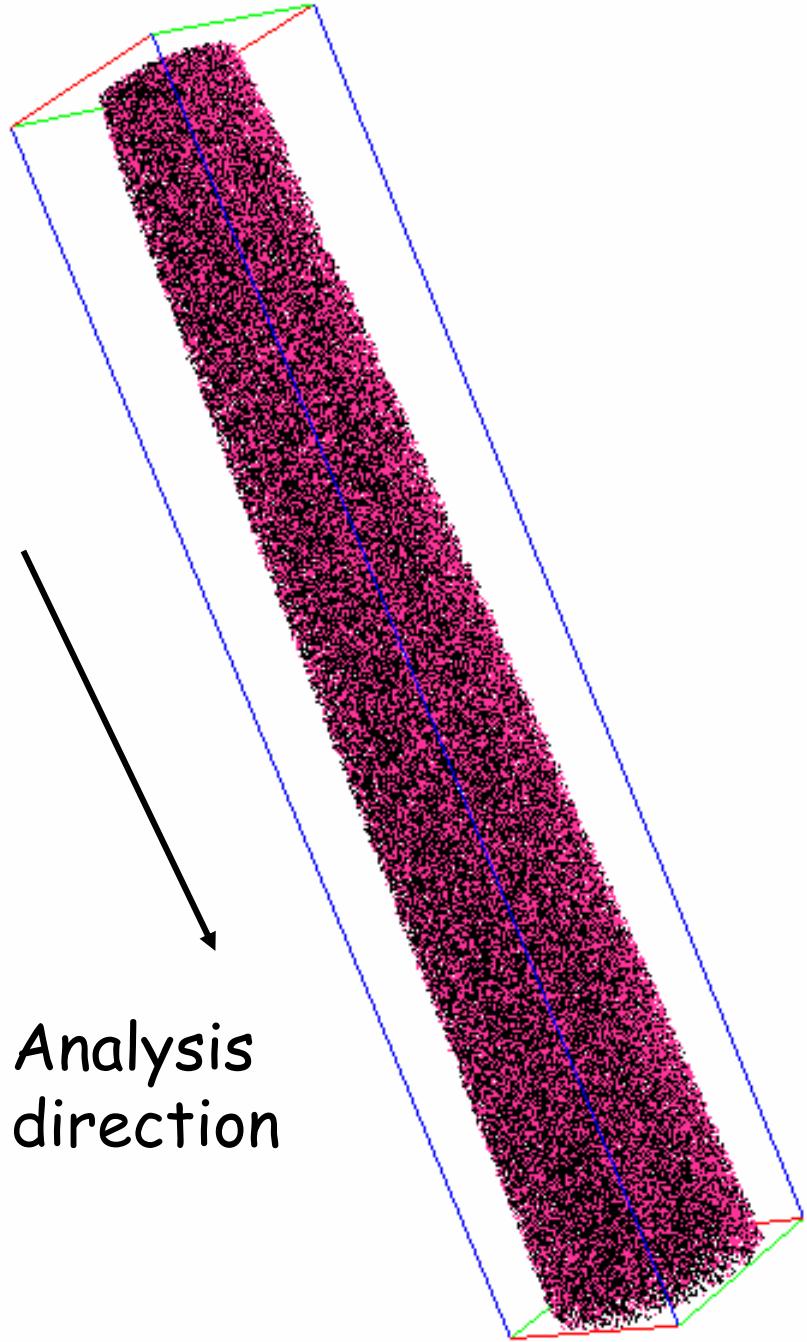
Mass Spectrum



- $O^+$ : 16  $H_2O^+$ : 18
- $Nb^{4+}$ : 23.25  $NbOH_6^{4+}$  /  $NbO_3H_3^{5+}$ : 29
- $Nb^{3+}$ : 31  $NbO^{3+}$ : 36.3
- $Nb^{2+}$ : 46.5  $NbO^{2+}$ : 54.5  $NbO_2^{2+}$ : 62.5
- $NbO^+$ : 109  $NbO_2^+$ : 125

# Proximity Histogram

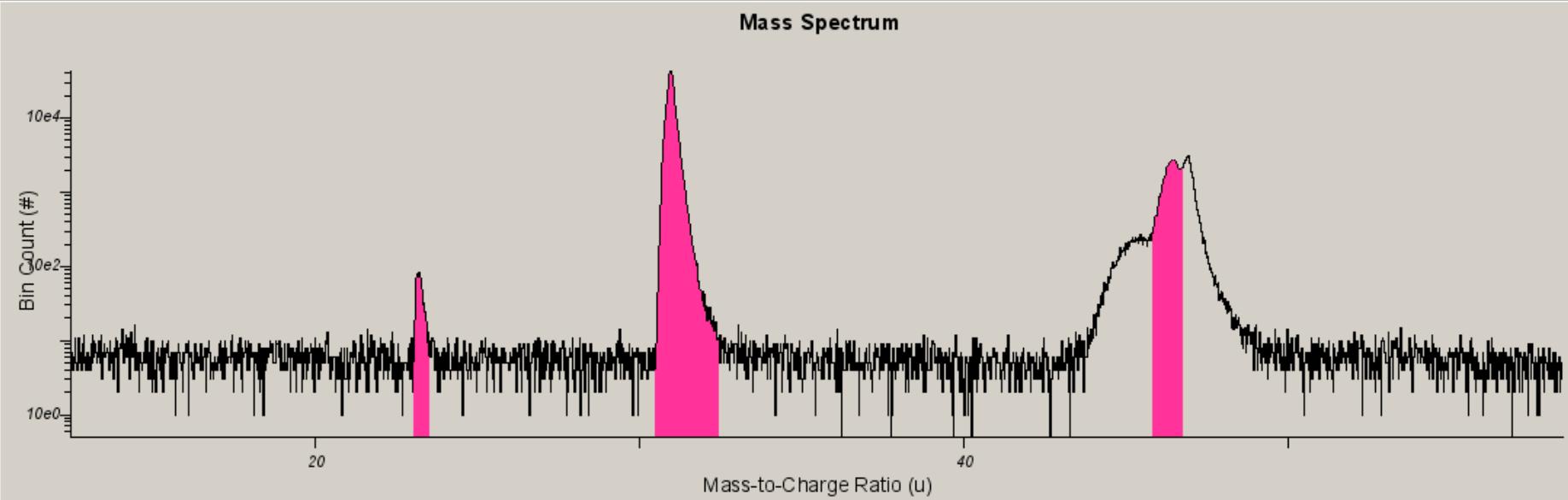




## 4<sup>th</sup> Results

- $21 \times 21 \times 123 \text{ nm}^3$
- 0.9 M atoms
- Nb magenta
- H black
- No O detected

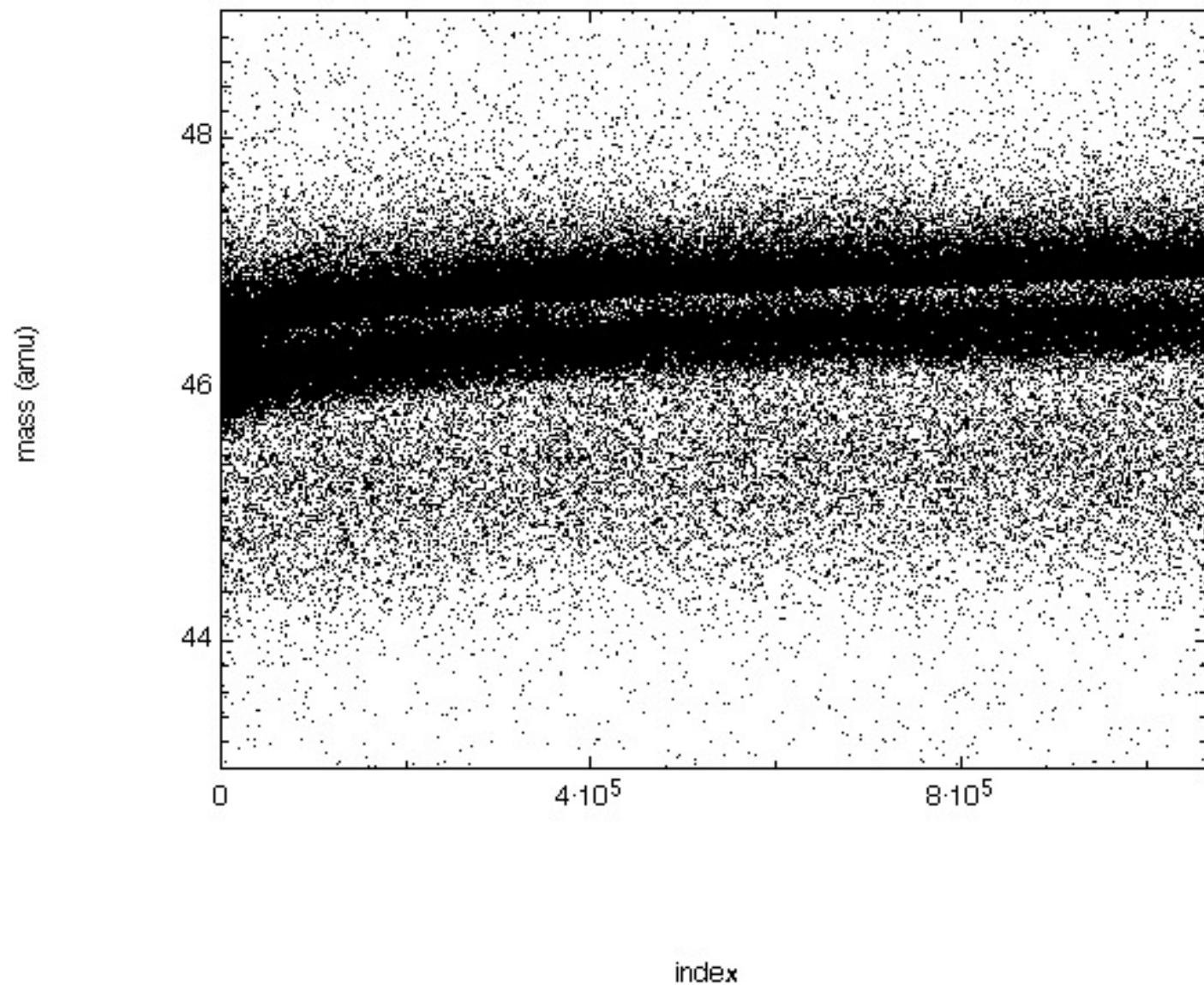
# Mass spectrum

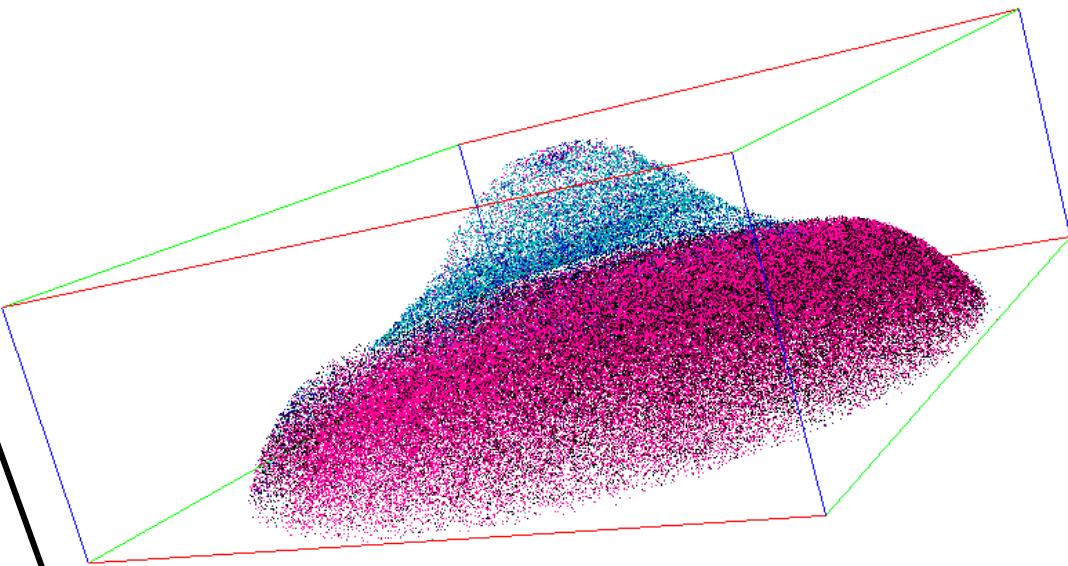


- $\text{Nb}^{4+}$ :23.25
- $\text{Nb}^{3+}$ :31
- $\text{Nb}^{2+}$ :46.5

# Mass chart

output.atomlist

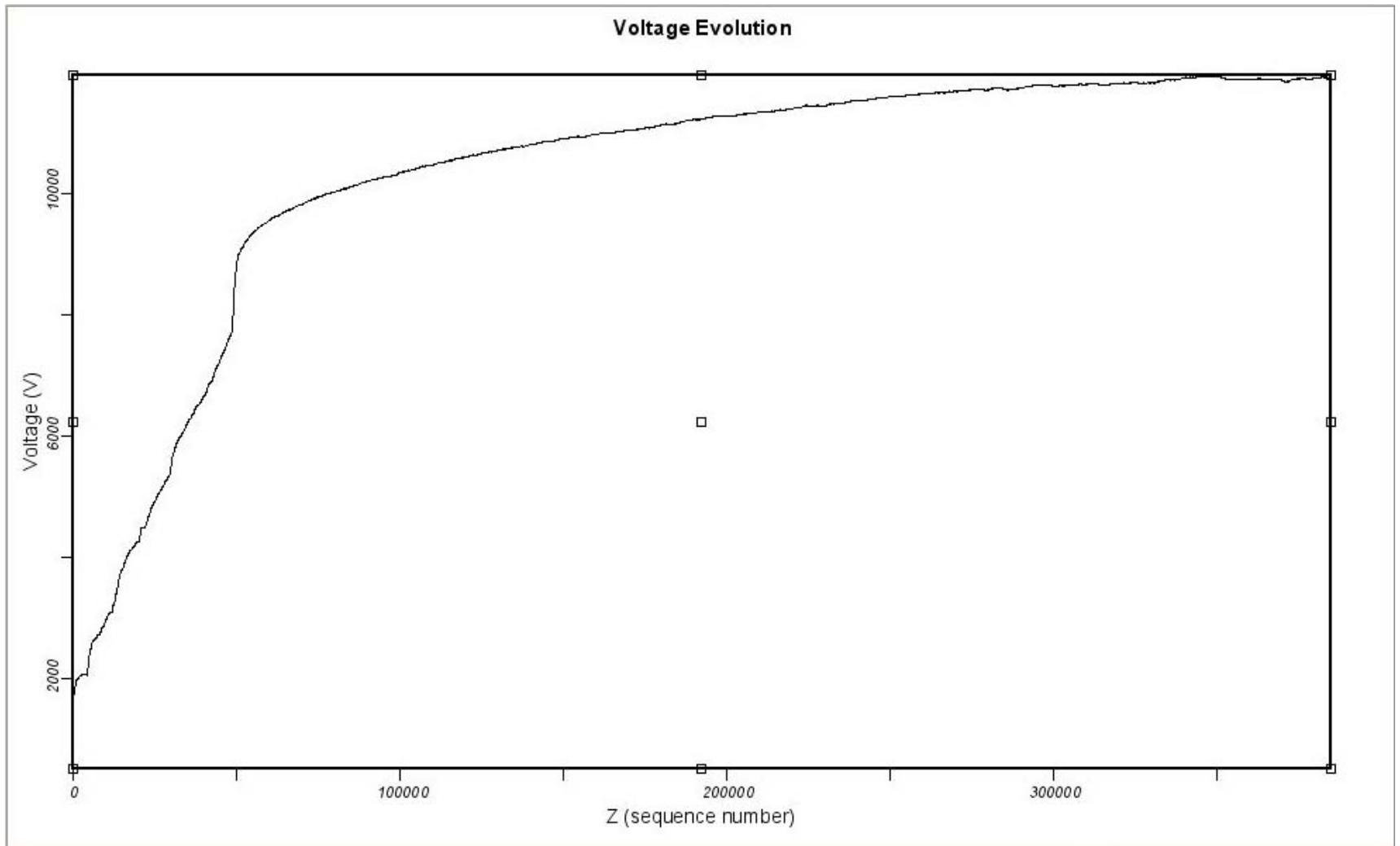




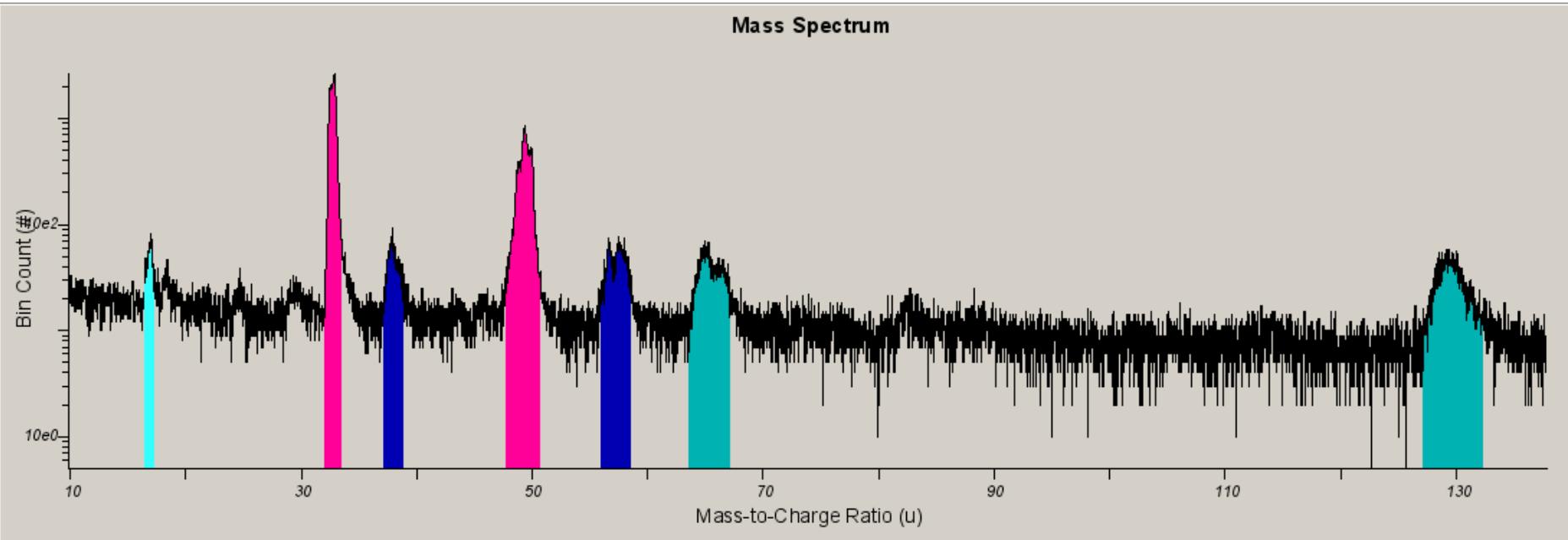
## 5<sup>th</sup> Results

- $21 \times 21 \times 123 \text{ nm}^3$
- 0.9 M atoms
- Nb magenta
- H black
- No O detected

# Voltage curve



# Mass spectrum



- $O^+$ : 16
- $Nb^{4+}$ : 23.25
- $Nb^{3+}$ : 31  $NbO^{3+}$ : 36.3
- $Nb^{2+}$ : 46.5  $NbO^{2+}$ : 54.5  $NbO_2^{2+}$ : 62.5
- $NbO_2^+$ : 125

# Summary

tip	oxide	interface	transition
Previous results	2 nm -Nb <sub>2</sub> O <sub>5</sub>	10 nm	smooth
	2 nm-Nb <sub>2</sub> O	8 nm	rapid
1st	15 nm-Nb <sub>2</sub> O <sub>5</sub>	15 nm	smooth
2nd	0 nm	0 nm	Bulk Nb
3rd	5 nm-Nb <sub>2</sub> O <sub>5</sub>	15 nm	smooth
4th	0 nm	0 nm	Bulk Nb
5th	N/A	N/A	

# Conclusions

- Atomic scale investigation of surface oxide to bulk Nb
  - First time to reach bulk Nb
  - Surface: possibly  $\text{Nb}_2\text{O}_5$
  - Thicker than expected:  $> 5 \text{ nm}$
  - High level of O below surface oxide:  $\sim 20\%$
  - High level of H in oxide and bulk:  $\sim 20\%$
  - Scattered results
    - Electropolishing dependent
    - Need better statistics

# Next step

- TEM investigation
  - Structure of oxide
- Use FIB for sample preparation
- Investigation of heat-treated Nb samples